Irish Interdisciplinary Journal of Science & Research (IIJSR)
Volume 7, Issue 2, Pages 71-83, April-June 2023

Dying Properties of Natural Dyes Extracted from *Parkia biglobosa* Tree Bark on Cotton Fabric

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DOI: https://doi.org/10.46759/IIJSR.2023.7209

Article Received: 12 April 2023 *Article Accepted: 20 May 2023* *Article Published: 03 June 2023*

ABSTRACT

The research work was carried out to extract natural dyes from Parkia biglobosa tree bark. Synthetic dyes are toxic, non-biodegradable and not friendly to the environment i.e. non-eco-friendly. Natural dye therefore perfectly fit into this since they are non-toxic, biodegradable, non-hazardous and more eco-friendly. Natural dye was extracted from Parkia biglobosa tree bark using Soxhlet extraction method and the dye extract was applied on cotton fabric. The percentage exhaustion, fastness properties and effect of mordant on the dyed samples was evaluated. The post-mordanting method was used to apply the mordant (potassium dichromate) on the samples. The dyeing of the samples were carried out based on the dyeing parameters such as dye concentration, electrolyte concentration, time and temperature respectively. From the result obtained, it was observed that the percentage exhaustion–quantity of dye absorbed by cotton fibre decreases with increase dye concentration. Similarly the absorption of dye was increased with the decrease of dye concentration. The highest percentage exhaustion of 96% was obtained at 10% dye concentration. However, it is observed, that increase in electrolyte concentration resulted increase in the percentage exhaustion. The highest percentage exhaustion of 61% was obtained at 10% electrolyte concentration. This observation is in agreement with the effect of electrolyte concentration on percentage exhaustion. From the result obtained, it was observed that the percentage exhaustion–quantity of dye absorbed by cotton fibre increases with increase in time of dyeing. The highest percentage exhaustion of 76% was obtained at 100 minutes dyeing time. It is obvious, from Figure 4 that the rate of dyeing increases gradually by raising the temperature. The highest dye exhaustion was attained at boiling (100^{o}C). It may be suggested that the extracted dye in the aqueous medium is in aggregated form since they need an increase temperature to migrate into the fibre. In the effect of mordant on dye uptake and enhancement of fastness properties was evaluated from the result; it was observed that mordanting have improvement on the fastness properties. Comparing with the sample without mordant, post-mordanting have a better fastness property. However, on the whole, the mordant sample performs better. From the results it can recommended that dye extracted from Parkia biglobosa Tree Bark can be used to dye cotton fabric. The post-mordanted treated fabric samples have improved fastness properties when compared with non-mordanted fabric samples. The mordant also changed the shade of the dyed samples.

Keywords: Fastness properties; Natural dyes; Mordants; *Parkia biglobosa*; Percentage exhaustion.

Introduction

Oforghor (2017), Misha and Singhal (2015) reported that since prehistoric time natural dyes is used for coloring of food substrate, leather as well as fibers like wool, silk and cotton. The use of non-allergic, non-toxic and eco-friendly natural dyes on textiles have become a matter of significant importance due to the increased environmental awareness in order to avoid some hazardous synthetic dyes (Agarwal, 2009).

Natural dyes extract from a variety of the substance with are occur in nature such as plants (e.g., indigo and saffron); insects (e.g., cochineal beetles and lac scale insects); animals (e.g., some species of mollusks or shellfish); and minerals (e.g., ferrous sulfate, ochre, and clay) without any chemical treatment. (Kasiri and Safapour, 2015) and Sara-Kadolph, 2008). In the earlier days, dyes were derived only from natural sources. Some processing was required but essentially the dye itself was obtained from a plant, mineral or animal. Oforghor (2011) reported that after the accidental synthesis of mauveine by William Henry Perkin in 1856 and its subsequent commercialization, heralding the advent of coal tar dyes (now synthetic dyes), the use of natural dyes receded.

A wide range of synthetic dyestuff was thrust upon the industry and it was readily accepted for its distinct advantage over natural dyes with respect to application, colour range and availability. Mathur *et al.* (2001) and Ibrahim *et al.* (2010) stated that the status of Natural Dyes in India has a very rich tradition of using natural dyes.

The art and craft of producing natural dyed textile has been practiced since ages in many villages by traditional expert crafts-persons in the country. Natural dyes, when used by themselves have many limitations of fastness and brilliancy of shade. However, when used along with metallic mordants they produce bright and fast colours. The use of metallic mordants is not always eco-friendly, but the pollution problems created by metallic mordants are of very low order and can be easily overcome (Nandal *et al.*, 2001 and Khan *et al.*, 2020). Oforghor *et al.* (2020) and Narbona *et al.* (2020) stated that dyes are substance which when applied to fabric surface are expected to adhere firmly, therefore, dyes are coloured substance that has affinity to the substitute to which it is being applied; it can also be defined as any natural or synthetic substance used to add a colour to or change the colour of something Natural dyes are types of dyes normally obtained from vegetables, plants parts, (leaves, root, and fruits) minerals and insects. These types of dyes were in use throughout civilization and up-till today are being used in some traditional/native clothes, jute fibres for making local bags and mats, but are no longer in used by modern dyeing industries such types of dyes include indigo, madder, ochre, catechu, saffron, henna, alizarin and animals dyes such as kermes, cochineal lac fish (Kansik *et al.*, 2002, and Repon *et al.*, 2017). The minerals dyes are Prussia blue, chrome-producing yellow, iron having buff (Hobhouse, 2001). However, the natural dyes have certain limitation such as longer time of extraction and dyeing; low color value and fastness; few types of color shades. Even though natural dyes have beauty bright fastness of colour on fabric, yet they are easily renewable, available, non-allergic to the skin, non-toxic and very compatible with the environment (Polette-Niewold *et al.*, 2007).

Alan *et al.* (2007), Ashis and Priti (2009), Bansal and Sood (2001) reported that *Parkia biglobosa* also known as African locust Bean (West African names: ogiri, enon, kuweahi ghena, talong, doruwa or iru) is a perennial deciduous tree of the family fabaceae, in the genes parkia. *Parkia biglobosa* is a dicotyledonous angiosperm belonging to the family fabaceae (caesalpinioideae-minosoid calede). It is categorized under spermatophytes, vascular plants, it is a deciduous perennial that grows to be between 7 and 20 meter high, in some cases up to 30 meters (Chan *et al.*, 2000, Chavan and Chakraborty, 2001 and Cris and Vilarem, 2001). The tree is fire- resistant halophyte characterized by a thick dark grey-brown bark. Robert Brown describes the genus *parkia* in 1826. He named it after Mungo Park, a Scot who made two remarkable journeys of exploration into the interior of west African in 1795 - 1797 and 1805 (Habibzadeh *et al.*, 2010, Jagatheeswari *et al.*, 2013). Husk of pod mixed with indigo improve the lustre of dye products. Barks and pods contains piscicides; the alkoloid parkine that occurs in pods and barks maybe responsible. Barks is used as a mouth wash, vapour inhalant for toothache, or for ear complaint. The tree of African locust Bean requires between 0-300meters altitude, a mean annual rainfall of between 400-700 millimeter and a mean annual temperature of about 24-28°C it prefers well-drained, thick clay soils but can also be found on shallow, thin Sandy soils (Saravanan *et al.*, 2013 and Acguah and Odura, 2012). They grow in the savanna region of West African up the southern edge of sahel zone 13° NE, according to Campbell-platt, 2000). Natural dyes cannot be used directly from the renewable source. Using raw material for dyeing has many limitations. Safe and cheap extraction of main colouring component is most important without affecting the extraction conditions and avoiding any contamination in various extraction techniques. Several extraction methodologies for natural dye that comply with both consumer preference and regulatory control and that are cost-effective are becoming mordant popular (Gulrajani, 2002). The word mordant comes from the Latin

word "*modere*" meaning to bite. A mordant is a chemical which can itself be fixed on the fiber and also forms Chemical bond with the natural colourants. It helps in absorption and fixation of natural dyes and also prevents bleeding and fading of colours i.e. improves the fastness properties of the dyed fabric (Oliver, 2010, Rangsriwong *et al.*, 2018 and Netherton and Gale, 2007). The broad objective of this study is the Dyeing and printing Properties of Natural Dyes Extracted from *Parkia biglobosa* Tree Bark on Cotton Fabric.

Experimental

Materials

100% white cotton fabric (purchased from Lafia metropolitan market), *Parkia biglobosa* barks (powder), Scissors, Beakers, Methanol (CH_3OH), Distilled bottom flask, Condenser, Thimble, Soxhlet extraction chamber, Measuring cylinder, thermometer, Syringe, Masking tape, Filter paper, Glass rod, Sample tube, Test tube, Funnel, Pencil, Ethanol, ruler, potassium dichromate, heating mantle, pipette, retort standard, covette, spectrophotometer, white plastisol ink and squeegee.

Method

Collection of plant materials

The *Parkia biglobosa* barks (commonly known as locust bean plant) were collected from Odu, in Aboshiyo Udege Development, Area, Nasarawa State, Nigeria.

Pulverizing

The barks were dried under shade and pounded into finely powdered using mortar and Pestle then sieved, and the finely powdered was poured in a plastic container and covered.

Extraction of dye

100g of dried barks powder (*Parkia biglobosa*) was packed and placed into a thimble of soxhlet extraction, 1000ml of Methanol was weighed and poured in a flat bottom flask and was fixed on the heating mantle, the soxhlet extraction chamber was fixed on flat bottom flask and a pipe was fixed at the water inlet (where water passes through the condenser) and another pipe was fixed into the water outlet (where passes out of the condenser) and the condenser was fixed to the soxhlet extraction chamber, tap was on and the heating mantle was on. The flat bottom flask was then heated gently, the Methanol boiled at 64.7°C which is the boiling point of methanol, the vapour in the flat bottom flask then leaves through distillation path and move straight to the condenser where it is being condensed back to the ground state, the extract move back to the flat bottom through the siphon tube when the solvent reaches the top the process automatically repeats itself. The extract was heated at low temperature in order to allow the solvent (methanol) to evaporate leaving the dye.

Paper Chromatography

Filter paper was cut into a strip that was about 2.2cm width and 22cm length, 2cm was measured and drawn at the bottom edge of the paper which is the starting line, a dot was marked at the starting line and the extract was spotted at the dot .10ml of methanol was measured and poured in a Measuring cylinder and the filter paper was put inside

and covered for 1hr. After 1hr it was removed and the distance moved by the solvent and distance moved by solute (extract) was calculated.

Importance of Retention factor

In chromatography, Rf values are the most basic prerequisite of the experiment. These numbers indicate whether the analyte (solute) prefers the stationary or mobile phase. With stationary and mobile phases, Rf values are used to determine, polarity, relative masses, and relative solubilities among other things.

The retention factor (Rf value) in chromatography is that it can be used to predict where a particular substance will be located on the chromatogram.

The Rf values serves as a simple measurement of the relative of the compound of interest under the experimental conditions was calculated using the following formula;

$$Rf = \frac{\text{Distance moved by solute}}{\text{Distance moved by solvent}} \quad (1)$$

Mobile phase=11.1 cm; Solute = 10.3 cm

$$Rf = \frac{10.3 \text{ cm}}{11.1 \text{ cm}} = 0.93$$

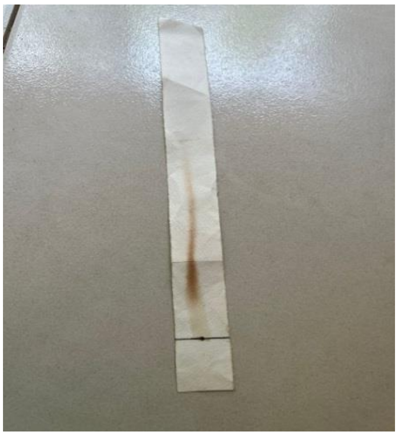

Plate 1. Paper Chromatography

Dyeing procedure

The cotton fabric samples were dyed with the extracted dye using the exhaustion method. 10% stock solution was prepared from plant extract and 3% acetic acid was prepared as an electrolytes. (6) test tube were introduced and the dye concentration and electrolytes were measured using a syringe at vary ml. 1g of cotton fabric was soaked with distilled water and was introduced to each test tube (6), the water bath was on and thermometer was placed inside the water bath, the fabric was introduced and dyeing commenced at 30°C, the temperature raised to boil during 10minutes, stirring of the samples was done at frequent intervals, the temperature of dyeing was at 90°C, it was maintained for 60 minutes after which the samples were removed and dried and the residual dye solution was poured in a sample tubes. The following variables concentration, electrolytes, time, and temperature were used for dyeing shown in Table 1,2,3,4. The volume required from each stock solution was calculated based on the formula;

$$\text{Volume}\quad \text{cm}^3 = \frac{WP}{C} \qquad (2)$$

Where:

P = percentage shade;

W = weight of fabric;

C = Percentage concentration of stock solution;

V = volume (cm^3).

Procedure for applying mordant

Mordanting was carried out using Post mordanting.

Fabric dyed with extracts and samples were then taken out of the dye bath, squeeze. Then (4% o.w.f) of potassium dichromate with the squeezed dyed sample without washing. This is done at 60°C for 30 minutes with M:L of 1: 20 (Oforghor *et al.*, 2016).

Measurement of the wavelength of maximum absorption of extracted dye

The method of Oforghor (2011), was adopted for measurement then the absorption spectra of the dye was measured with the aid of UNICOM SP800B spectrophotometer in the wavelength range of 400-800nm in a glass cell of path length of 1cm, the measurement of the spectra was made at a concentration of 0.95g/l in ethanol, with ethanol as reference. The wavelength of maximum (UV-Vis) was found at 450nm.

Determination of optical density (% exhaustion)

The optical density of the extracted dye was determined before and after dyeing at the maximum wavelength of (450nm) of each dye using spectrophotometer, the percentage exhaustion was calculated using the expression below:

$$\text{Percentage exhaustion} = \frac{ODB - ODA}{ODB} \times 100 \qquad (3)$$

ODB= optical density before dyeing; ODA =optical density after dyeing.

Washing Fastness

2g of sanitol soap/100ml was dissolved in cool water and was stirred until it became homogeneous solution, the samples of the dyed fabric were added and squeezed gently for 5 minutes, and dried. The dyed fabric was assessed using grey scale.

Colour Measurement

This assessment is based upon the magnitude of visual contrast between the original (untreated) 100% white cotton fabric and the tested sample. It was assessed using the Grey scale for colour change and staining. The Grey scale of assessing change in colour consists of five (5) pairs of pieces of colour chart. The staining was assessed using the

grey scale for assessing stain. The scale consist of nine (9) pieces of colour chart 5,4-5,4, 4,3-4, 3, 2-3, 2,1, were six (6) is a pair of white six pieces of cotton fabric giving a series of contracts increasing in geometric progression.

Screen Printing

White plastisol ink was mixed with the extracted dye to form paste. The screen was made of a piece of mesh stretched over a frame, a stencil is formed by blocking off part of the screen in the negative image of the design to be printed, that is, the open space where the ink is appear the substrate. Screen was prepared by applying emulsion ink and was allowed to dried before exposing to UV light, the screen (mesh) was placed on top a substrate, the paste was placed on of the screen and squeegee (rubber blade) was used to push the paste the through the open areas of the mesh.

Results

The effect of dyeing parameters, which are dye concentration, electrolytes, time and temperature, washing fastness and mordanting were ascertained.

Table 1. Effect of increasing dye concentration on dye absorbance

Concentration	ODB	ODA	% exhaustion
2 ml	0.773	0.014	96
4 ml	0.773	0.094	89
6 ml	0.773	0.167	78
8 ml	0.773	0.182	76
10 ml	0.773	0.274	65
12 ml	0.773	0.399	48

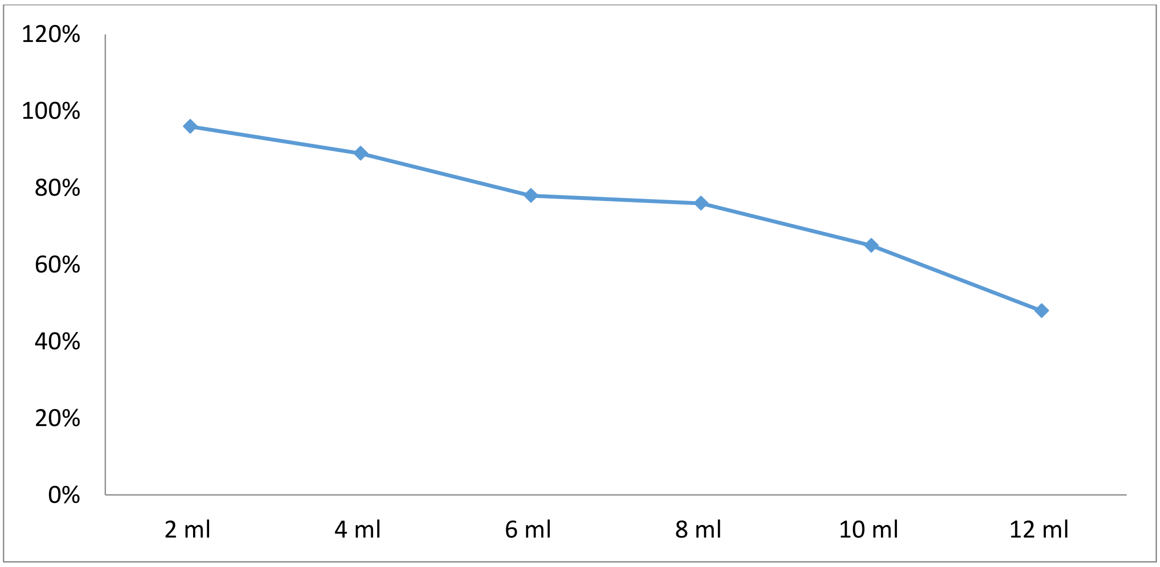

Figure 1. Percentage exhaustion of extracted dye concentration of *Parkia biglobosa* bark

Table 2. Effect of increasing dye electrolyte on dye absorbance

Electrolyte	ODB	ODA	% Exhaustion
0 ml	0.773	0.399	48
2 ml	0.773	0.274	68
4 ml	0.773	0.226	70
6 ml	0.773	0.229	70
8 ml	0.773	0.094	89
10 ml	0.773	0.095	89

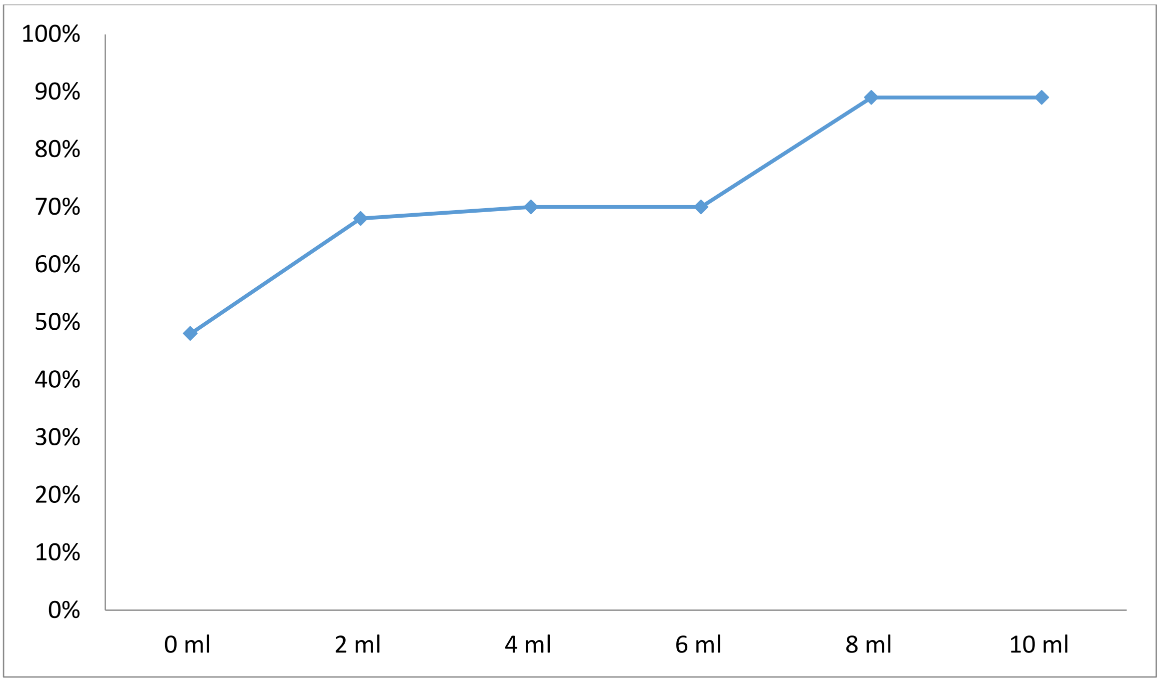

Figure 2. Percentage exhaustion of extracted dye concentration of *Parkia biglobosa* bark

Table 3. The effect of increasing dye time on dye absorbance

Time (Hours)	ODB	ODA	% Exhaustion
0 h	0.773	0.182	76
1 h	0.773	0.167	78
2 h	0.773	0.078	89
3 h	0.773	0.041	94
4 h	0.773	0.014	94
5 h	0.773	0.014	94

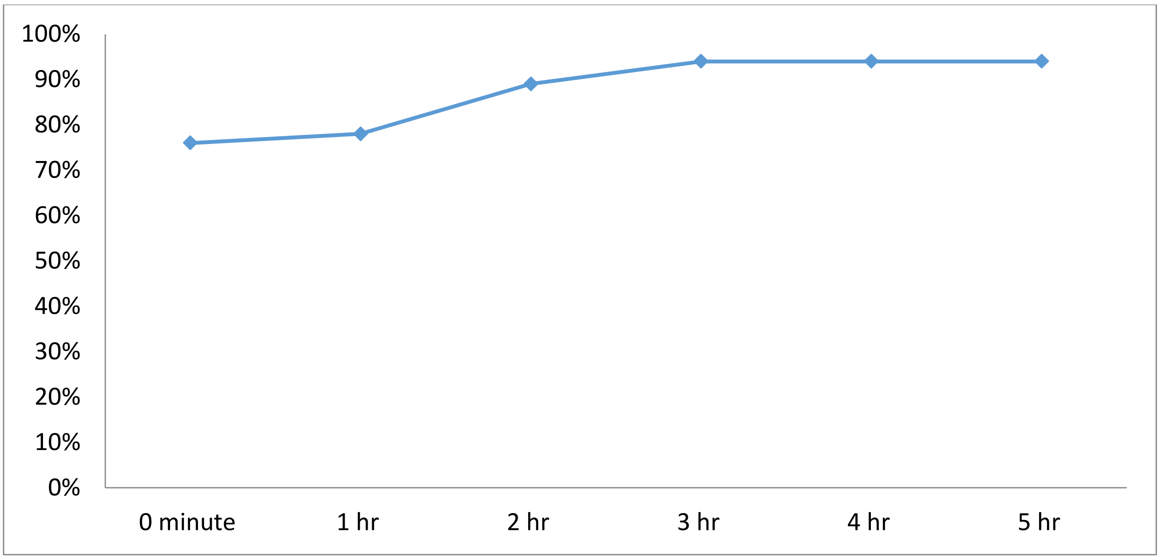

Figure 3. Percentage exhaustion of extracted dye concentration of *Parkia biglobosa* bark

Table 4. Effect of increasing dye temperature on dye absorbance

Temperature	ODB	ODA	% exhaustion
30^0 C	0.773	0.512	30
40^0 C	0.773	0.426	44
50^0 C	0.773	0.461	53
60^0 C	0.773	0.274	64
70^0 C	0.773	0.135	88
80^0 C	0.773	0.136	83

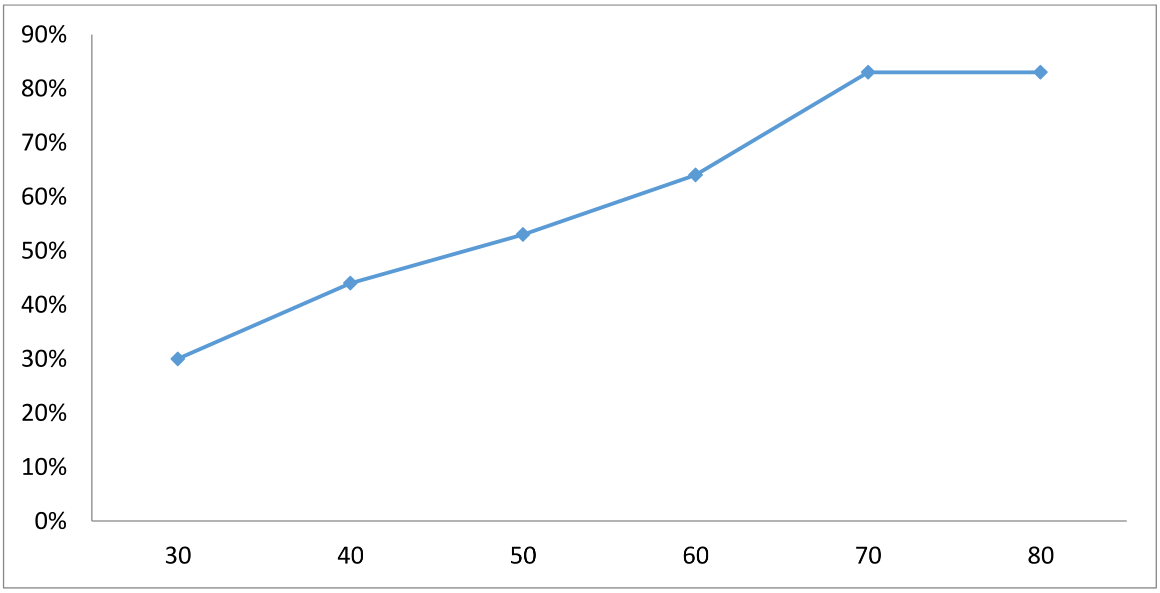

Figure 4. Percentage exhaustion of extracted dye concentration of *Parkia biglobosa* bark

Table 5. Effect of washing fastness on the dyed sample

Non mordanted Samples	A. *Colour change*	Colour of dyed samples
Concentration 2ml	3	Light-brown
Electrolyte 2ml	3	Light-brown
Time 4h	3	Light-brown
Temperature 70°C	4	Deep-brown

1. Very poor; 2. Poor; 3. Fair; 4. Good or Moderate; 5. Excellent

Table 6. Effect of mordant on the dyed samples

Mordants	Mordanted sample	Colour change	Colour of mordanted sample
Potassium dichromate	Concentration (2ml)	5	Cork
	Electrolytes (2ml)	5	Cork
	Time (1h)	5	Cork
	Temperature (30°C)	5	Cork

Discussion of Results

It is observed that the dye absorption by cotton fabric decrease with the increase of dye concentration in the dye bath. This may be explained that the presence of more dye ions hinder absorption of dye to fabric, where rare ions favour it. It is to be mentioned that with the increase of dye concentration, the absolute quantity diminished. The size of dye particles in solution usually depends on temperature, concentration of electrolyte and concentration of dyes (Oforghor, 2017). The size of dye particles always increased with an increase in dye concentration.

At equilibrium a more or less pronounced selective absorption of the dye by the fabric is observed up to full exhaustion of the dye bath. Force of interaction of the dye and the dye bath. The exothermic reaction taking place at dyeing confirm the occurrence of the interaction. From the experiment it was observed that bright and uniform shades were produced when cotton fabric was dyed with 8ml dye solution. Above and below these percentage of dyes dull and uneven shades were obtained.

The effective concentration of dye e.g. 8 ml of concentration corresponding to 76% dye exhaustion from the test. The behavior of dye absorption in the dyeing of cotton fabric depends upon the characteristic of dye towards the fabric. It is observed that 48% of dye is absorbed from the test tube containing dye without electrolytes in the test

tube i.e. zero concentration of electrolyte. This is attributed to the fact that in the absence of electrolyte, dyes have a poor affinity towards the fabric due to the presence of similar charge group between the fabric and the dye.

It is also observed that the absorption of dye increased with the increase of electrolyte concentration in the dye to saturation absorption and beyond this, no change in absorption of dye occurs on further addition of electrolyte. The experiments showed that the absorption reaches saturation absorption when cotton fabric is dyed in the presence of 8ml as electrolyte and the corresponding dye exhaustion is 89%, above or below this Percentage of electrolyte shades were uneven and dull. The behavior of dye absorption depends upon the characteristic of dyes towards the fabric.

It is observed from the experiment that the longer the Dyeing time the higher the dye exhaustion, until the dye exhaustion attains equilibrium and there is no significant increase after further increase in dyeing time.

The effect of temperature on percentage dye exhaustion. It was observed that the absorption of dye cotton fabric increased with the increase of Dyeing temperature.it seems that the dye exist in solution as an aggregate of various when the temperature of the solution increase the thermal energy coming from inside impeded aggregation, and therefore, increase in the amount of the non-aggregate dye properties in solution.

The exhaustion of dyeing increases rapidly with the increase of temperature and reaches maximum when dyeing temperature is about 80^{o}C. The absorption of dye from the test tube at equilibrium dyeing temperature is 83%. The absorption remains same on further increase of temperature.

Conclusion

The present work shows that the barks of *Parkia biglobosa* can be used as dye for dyeing cotton fabric. The plants are grown through Nigeria and it is easily available plant. The whole process of extraction is eco-friendly. Different shades of colour can be obtained using different mordant. The dyeing extract has shown a change in hues when the mordant was added, the dye has good scope in the commercial dyeing of cotton. The mordant also shows a huge improvement in the fastness properties of the dyed samples as compared with the samples without mordants.

Recommendations

Based on the results obtained from this study, the researcher has the following recommendations to make:

- The clothing and textile section in secondary schools, colleges of education, and Universities can use the result of the study as a resource material for teaching creative skills in dyeing fabrics, fibers, tie and dye, batik and printing.
- The study should be used to educate students on the use of dye that is eco-friendly.
- The benefits of studying plant dyes and their applications in fabrics will improve the socio-economic value in the economy.
- Natural dyes are hypoallergenic, which means they are less likely to cause any allergic reactions when skin is exposed to them. This is ideal for those with sensitive skin conditions such as eczema, as well as babies and children.
- Natural dye has a good scope in the commercial dyeing of cotton.

Plate 1. Dyed Samples

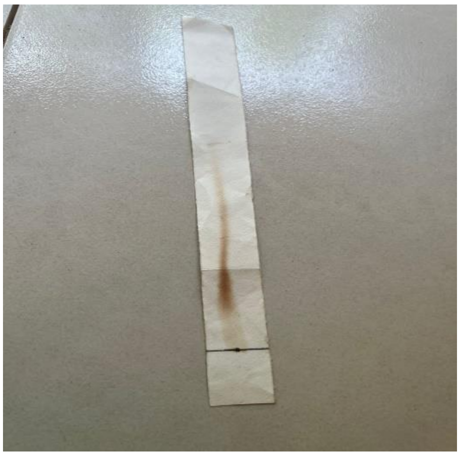

Plate 2. Paper Chromatograph

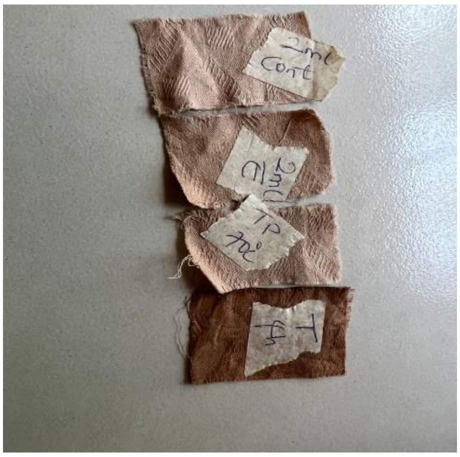

Plate 3. Washed Samples

Plate 4. Mordanted Samples

Plate 5. Screen-printed Samples using Natural Dyed

Plate 6. *Parkia biglobosa* (locust beans plant)

Declarations

Source of Funding

This research did not receive any grant from funding agencies in the public, commercial, or not-for-profit sectors.

Competing Interests Statement

The authors declare no competing financial, professional, or personal interests.

Consent for publication

The authors declare that they consented to the publication of this research work.

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